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Real Time Quantification of Activated Carbon Adsorption of Butanol and Evaluation of Influencing Adsorbent Characteristics

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Real Time Quantification of Activated Carbon Adsorption of Butanol and Evaluation of Influencing Adsorbent Characteristics

By:

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Date

Thesis Advisor

Chairperson of Department

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Abstract:

This paper uses a real-time data collection technique to analyze VOC adsorption onto activated carbon. Specifically, butyl alcohol is run through a column of extruded or granular activated carbon until it reaches an equilibrium concentration. These results are measured by a Photo-ionization Detector (PID) and reported to a computer for easy processing. Twenty-one total types of carbon were tested for capacity and time to breakthrough, with breakthrough being designated as 350 ppm as reported by the PID. Upon analyzing results of these tests, it is confirmed that surface area is proportional to adsorption capacity. It is also found that density is inversely proportional to adsorption capacity. Some of the carbons tested were treated with 0.1 Molar strong acid or strong base and allowed to dry. For every carbon treated this way, butanol capacity dropped by between 72 – 91% because of the destruction of the micropores.

1. Introduction

Volatile organic compounds, or VOCs, are a major source of air pollution. They are generated from natural and anthropogenic sources, and they can be a wide variety of molecules. Breathing in VOCs is not always harmful, but in higher concentrations or with more toxic molecules, serious negative health effects are possible. One commonly used way of removing VOCs from the air is activated carbon treatment. Activated Carbon, or AC, is a porous material with extremely high surface area. VOCs will stick to the surface of the AC, because they are more attracted to the surface functional groups than to the air[1].

Activated Carbon can be made from hard materials primarily composed of carbon. Common materials used to make AC are coconut shells, coal, wood, and previously used activated carbon [2]. These materials are heated in an oxygen depleted environment, so that the carbon does not combust. Any volatile compounds will escape the underlying structure, leaving behind a solid structure made almost entirely of carbon that has high surface area and complex pore structure. Three common types of AC are powdered activated carbon (PAC), granular activated carbon (GAC), and extruded activated carbon. PAC is used primarily in batch water phase applications, because flowing a fluid through a packed bed of it would cause too much pressure drop. GAC is used in both air and water phase applications, but can vary widely in particle sizes [3]. Extruded carbons are manufactured to be pellet shaped, and are used almost exclusively in air phase remediation systems because they are larger and have lower pressure drops. This paper uses only GAC and extruded carbons.

VOCs adsorb onto activated carbon because of interactions between the organic molecule and the carbon surface. Because of this, different VOCs will have different adsorption capacities and adsorption kinetics. This paper uses butanol, which is a four-carbon alcohol molecule. The OH functional group on this molecule induces a dipole giving its ends partial charges. Depending on the surface properties of the carbon being used, this can either help or hurt the adsorption capacity, which is the main variable of interest for this study. Molecular weight is another variable that greatly affects adsorption. Higher molecular weight compounds tend to adsorb better than lower ones, because they are more ready to leave the air phase, and because they have a lower surface area to volume ratio. This means that while the particle is bigger, the amount of surface area it takes up on the carbon will be less. Butanol is relatively small, and therefore does not adsorb as well as some other, larger compounds.

Removal of alcohols from air is an important subject because indoor air pollution, which typically has lower concentrations, is often dominated by alcohol cleaners and solvents. Small alcohol molecules adsorb most efficiently on carbons with high proportions of micropores, and frequently form a bilayer on the carbon surface[4]. Activated carbon is also easy to reuse when alcohols are adsorbed to it [5]. Just by running clean air through it, more than 98% desorption or recovery can occur [4], [6]. The heat of desorption is greater than the heat of vaporization for butanol, which suggests a reaction between butanol and the surface functional groups on activated carbon [6], [7], [8].

Adsorption of VOCs onto activated carbon should be repeatable for the same compound, carbon type, and temperature. The isotherm and adsorption kinetics of each experiment were confirmed by running each test multiple times. There are four main types of adsorption isotherms, all of which were seen, will be talked about in the results and discussion section. However, the most common isotherm seen is the “S” isotherm. One of the main factors that determines isotherm parameters is the particle size distribution [9]. A test was done on a GAC that had a large distribution of particle sizes to confirm that channeling was not affecting the isotherm, and to verify that particle size did not affect adsorption capacity.

This paper looks to determine the properties of different activated carbons that yield the best butanol removal. Several carbons from four manufacturers were tested, and some were treated with strong acids/bases to see how it affected capacity. Some variables of interest are the surface area, density, and base material of the carbon. The manufacturers all provided this information themselves, and were not expressly tested for in the experimental procedure. The use of real time data collection allows the user to determine the quality of data shortly after a test begins, rather than at the end of the very long and time consuming testing process.

2. Materials and Methods

2.1: Activated Carbons

Activated carbons used in this paper came from Cabot, Calgon, Evoqua, and Nuchar. Three carbons came from Evoqua, three came from Cabot, five came from Calgon, and four came from Nuchar. Evoqua VCP 60 is an extruded carbon that is derived from anthracite coal [10]. Also used were two coconut shell based carbons, Evoqua 1230C and

1240Cat, which are both water phase carbons [11], [12]. They were used for comparison purposes; however, pressure drop data for them is only provided for water flows, and is therefore irrelevant for the air phase [10]. The 1240Cat is catalytic to oxidation/reduction reactions allowing contaminants to be broken down instead of adsorbed [13].

Cabot provided NORIT R2030 and SORBONORIT B4, are both extruded carbons and NORIT GAC 1240Plus is a granular activated carbon. The extruded carbon's specific base materials cannot be provided because it is proprietary information [14]. R2030 has a surface area of 800 square meters per gram, and SORBONORIT B4 has 1250 meters squared per gram [15], [16]. The 1240Plus is derived from Bituminous Coal and is intended for use in water applications [17].

Calgon provided their OVC 4X8, VPR 4X10, BPL 4X10, and 207C 6X12 granular activated carbons, as well as their AP460 extruded activated carbon. 207C 6X12 and OVC 4X8 are both made from coconut shells [18], [19]. BPL 4X10 and AP4-60 are both made from bituminous coal [19], [20]. Lastly, VPR 4X10 is made from reactivated carbons [21].

Nuchar, owned by Ingevity, provided their BAX 1500, and BAX 1100, both extruded carbons. They also provided their WV-A 1500 and WV-A 1100 granular activated carbons. All four carbons provided by Nuchar are chemically activated carbons with a wood base [22]. BAX 1500 and WV-A 1500 both have surface areas of 2000 - 2200 square meters per gram [23]. The WV-A 1100 and BAX 1100 have 1500 - 1700 and 1100 - 1300 square meters per gram respectively [24].

Acid and base treated activated carbons were made from samples of three regular carbons, placed in a bath of 0.1 M sulfuric acid, or sodium hydroxide. These carbons soaked in the acid/base bath for 24 hours, then were drained with a filter funnel. The remaining sample was placed in the oven and allowed to dry for several days. Each day the sample was weighed to see how much mass of water was lost. This continued until no more mass left the sample after 24 more hours of drying.

2.2: Apparatus

In order to test these carbons, an apparatus was created to deliver contaminated gas through a column and then be measured. The apparatus starts with compressed shop air, that flows through an air pressure regulator to make sure the pressure stays constant independent of what is coming from the source. From the regulator, the gas flows to a polyethylene bottle with a customized cap. The bottle contains the contaminant of interest (butanol in this case) and the cap has been fitted with tubing to allow clean air in, close to the contaminant surface. It also has been fitted with an exit tube so the contaminated air can move along through the system due to pressure in the bottle. Both fittings are sealed with an excessive amount of silicon sealant, so no leakage of contaminated air occurs. From the bottle, flow is tubed to a Bronkhorst USA Inc. EL-FLOW select mass flow controller. This allows for reliable and precise flow to the AC column. The column itself is made from two end pieces with a screwing mechanism, and a hand cut piece of one inch diameter clear PVC tubing. The ends were spiralized so that it can easily screw into the end pieces. The column is filled with carbon by placing it between two pieces of glass wool to hold it in place. From the column, the gas flows to an Ion Science TVOC photoionization detector (PID) to give concentration measurements in parts per million. A National Instruments

DAQ system is connected to the TVOC in order to record the data with LabVIEW on a nearby computer. After the gas concentration of butanol is measured, the flow is tubed to a fume hood. A diagram of the apparatus is provided below as Figure 1.

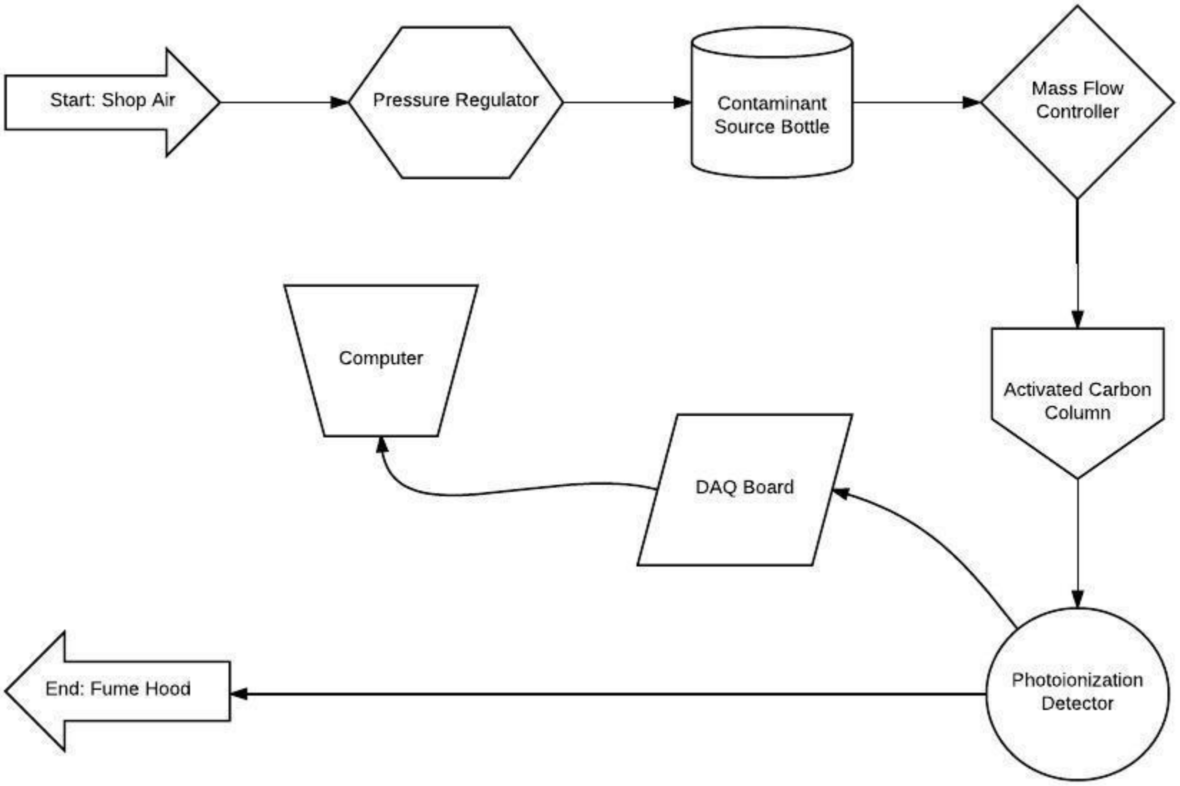


Figure 1: Process Flow Diagram of Apparatus

2.3: Experimental Operation

Before a test is conducted, various setup procedures must be completed. First of all, any carbon sample that is to be tested must be appropriately dried. This is done by placing over 30 grams of carbon into an oven at 100C and leaving it alone for at least 24 hours. Then, to cool the carbon without allowing atmospheric vapor to condense on and in it, the sample is placed into a desiccator. The sample will cool to room temperature within a few hours, however, the samples can be left in the desiccator for longer if need be. 10.0 grams of the dried sample is then placed into the clear PVC column and held in place by a small amount of glass wool. It is important that the sample is well packed, otherwise channeling will affect the shape of breakthrough and therefore the capacity at breakthrough.

Before the prepared column can be placed into the apparatus, it is wise to make sure the PID is working properly. Using a clean bottle and a blank column, shop air is allowed to flow through the system. The PID should read 0 or 1 ppm. If it doesn't, there might still be some residue from the previous test somewhere in the system. The shop air should flow until the PID reads 0 or 1 ppm before the next test is started. It is recommended that this process be started right away, while other parts of setup are completed.

Once the PID is ready, the carbon column is inserted into the system carefully, so as not to loosen the packing. Next, fill a contaminant bottle with 50 mL of liquid butanol. The shop air is then temporarily shut off at the source, and the clean bottle is switched with the filled one. Restart the airflow and click start on the LabVIEW program to begin data collection. Depending on the carbon, a test can take between two and five hours. Once breakthrough is reached, the test can be stopped electronically by pressing "Stop and Save" on the LabVIEW program after picking a file save destination and notepad file to be overwritten. Make sure to take note of the maximum concentration displayed on the TVOC before shutting the system down. This will be used as the equivalent concentration for calculations later.

Once the data is saved, proper shutdown must take place. First, shutting off the air temporarily will allow the contaminant bottle to be switched with the clean bottle once again. Switching the column with a blank one and resuming air flow will help save time before the next test, by removing butanol residue from the system. Dispose of the waste carbon and leftover butyl alcohol safely. Unplug the PID to save the life of its detection lamp. Once again, turn off the air flow, wait for flow to stop, set the mass flow controller's rate to zero, and stop communications with it. The mass flow controller can then safely be unplugged and the computer can be turned off.

2.4: Data Analysis and Calculations

The notepad file selected on LabVIEW will contain the data from the most recent test run, and can be copied and pasted into Microsoft Excel or a similar program for processing. When graphed, it will show a complete breakthrough curve, but will not yet give capacity or exact time of breakthrough. To get capacity, a MATLAB code was written to read an Excel file, and convert the data to capacity by taking the time difference between each step and the difference between the equivalent concentration and the concentration at each step, converting to mg of contaminant, and summing each step. This will give total mass adsorbed, and simply dividing by 10 will yield capacity of the carbon since every test was conducted with 10 grams of AC.

3. Results and Discussion

3.1: Summary of Data

Throughout this project, 15 different carbons were tested for capacity and three of those were treated with strong acids and bases. A comprehensive table showing capacities and breakthrough times is shown below as Tables 1 and 2. Intuitively, the more of butanol the carbon can adsorb, the longer the time to breakthrough is. Figure 2 is a graphical representation of Table 1 and Table 2, and shows capacity vs breakthrough for every carbon tested. It also shows by shade what base material each carbon was made from, and by shape what type of carbon it is (i.e. GAC, extruded, or meant for water applications).

Brand	Carbon	Capacity @ Breakthrough	Time to Break secs	Base Material	Density g/L	Surface Area m^2/g	C Cl4 #	Mesh Size
Evoqua		mg/g	seconds				or (2.55 * Butane #)	
	VCP60	300.0	9194	Anthracite Coal	500	Unavailable	60	4x6
	1230C	312.0	8458	Coconut shell	490	1100	NA	12x40
	1240Cat	156.0	8461	Coconut shell	490	1000	NA	12x40
Cabot								
	NORIT R2030	136.5	5280	Proprietary	520	800	16	4x10
	SORBONORIT B4	321.5	9111	Bituminous Coal	400	1250	70	4x6
	NORIT GAC 1240+	371.0	7273	Coal	500	950	NA	12x40
Calgon								
	207C 6x12	198.0	4478	Coconut Shell	500	Unavailable	55	6x12
	OVC 4x8	278.0	5525	Coconut Shell	450	Unavailable	60	4x8
	VPR 4x10	295.5	5711	Reactivated carbon	500	Unavailable	54.6	4x10
	BPL 4x10	327.0	6479	Bituminous coal	440	Unavailable	59.4	4x10
	AP4-60	216.0	5496	Bituminous coal	490	Unavailable	60	4x6
Nuchar								
	BAX 1500	756.3	15774	Wood	290	2100	28.8	6x10
	BAX 1100	407.0	12292	Wood	320	1200	29.3	6x10
	WV-A 1500	791.0	21184.5	Wood	280	2100	39	10x25
	WV-A 1100	621.5	17249.5	Wood	280	1600	28.8	8x35

Table 1: Summary of Carbon Performance

Some of the information on surface area and carbon tetrachloride number (the column labeled C Cl4 #) were not provided by the manufacturers. Cabot’s data sheet for NORIT GAC 1240+ mentions a coal base material, but does not say what type of coal it comes from. The base material of Cabot NORIT R2030 is proprietary. Capacity and time to breakthrough are the only measured values in this experiment. The manufacturers report density, surface area, carbon tetrachloride number, and mesh size directly. Since the surface area and density could easily change during the treatment processes used, all that is known for the treated carbons is the capacity and time to breakthrough. The mesh size remained roughly the same for each, so pressure drops would remain unchanged for the carbon beds. Table 2 shows the summary of data collected for treated carbons. Like the untreated carbons, multiple tests were conducted for each treated carbon, and the average values are reported.

Brand	Carbon	Capacity @ Breakthrough mg/g	Time to Breakthrough seconds
Evoqua	VCP 60	300.0	9194
	acid treated	35.2	3766
	base treated	52.5	4462
	1230 C	312.0	8458
	acid treated	73.9	3788
	base treated	86.9	3487
Cabot	SORBONORIT B4	321.5	9111
	acid treated	27.7	2546
	base treated	34.6	2743.5

Table 2: Summary of Treated Carbon Performance

Table 2 clearly shows large decreases in capacity for the treated carbons. This is even more apparent in Figure 2, where all 6 treated carbons are by themselves at the lower left hand corner of the graph. It was noted during preparation of the carbon columns that the 10 grams of treated carbons filled less volume than when untreated, even though they were dried for several days. The loss in carbon capacity is partially due to water infiltrating and staying in the remaining micropores [25]. In very small pores, water can stay adsorbed to the AC surface and would require much more energy to dry than our oven provided [26]. The lower capacity of the acid treated carbons is also due to higher affinity of water onto the surface than butanol for some functional groups [27], [28]. The acid oxidizes the surface more than the base, causing water to be slightly more attracted to the surface in the pores and take up more bonding sites [29].

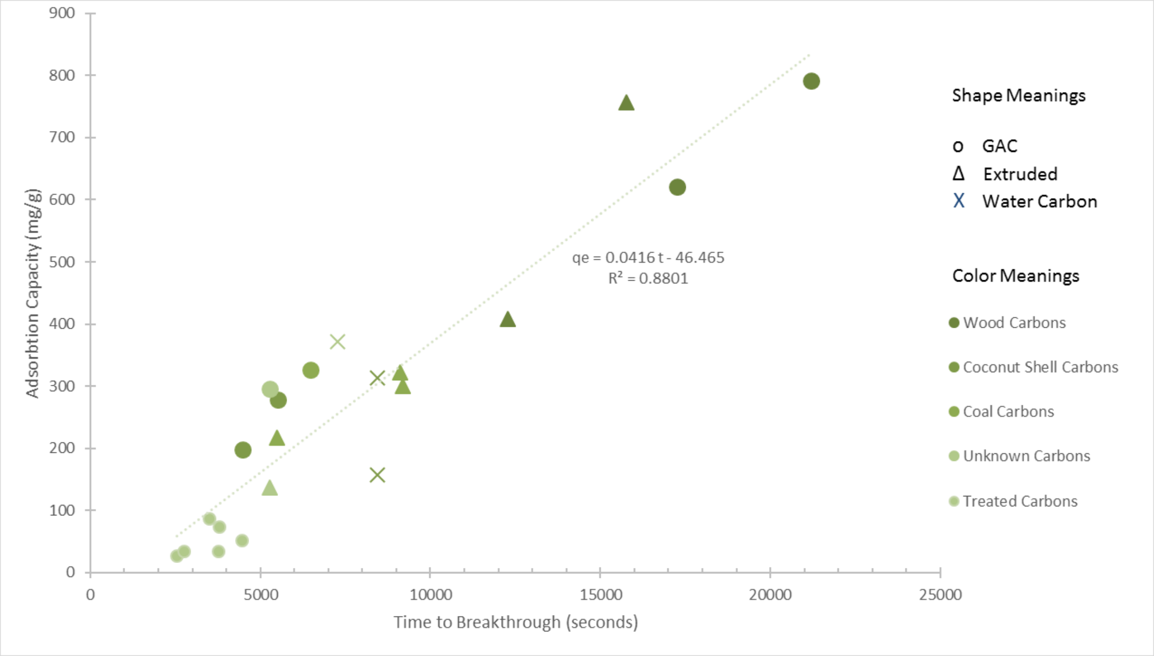


Figure 2: Graphical Representation of Carbon Performance Summary

The wood based carbons had the highest capacity, and any treated carbons had the lowest capacity at breakthrough. The other sources of carbon are all more closely related to each other. Breakthrough time for extruded carbons is generally lower than for GAC because of the shape of breakthrough and will be discussed in section 3.4. Note that Figure 2 has a trendline relating capacity and time to breakthrough. This line is representative of all data points shown in the figure.

It is well documented that adsorption capacity of activated carbons is proportional to surface area [30]. Figure 3 shows the results of capacity plotted versus surface area. As expected, the higher the surface area is, the higher the capacity is. This figure excludes the treated carbons, because after treatment, the surface area is likely reduced due to the destruction of the micropore structure and is therefore unknown. The relationship between surface area and capacity is linear, and has a high regression.

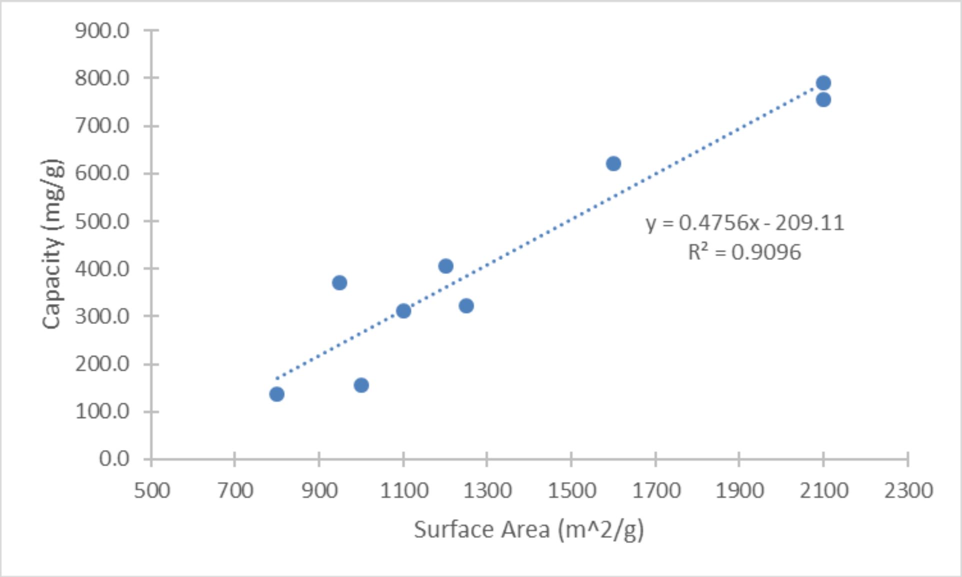


Figure 3: Adsorption Capacity vs Surface Area as Reported

The variable that relates to capacity that is not as well documented is density. Figure 4 shows the results of capacity vs bulk density of carbon. Again, this figure excludes the treated carbons because density for them is unknown. In this case, the carbons with lower density have higher capacity, likely meaning they have more total pores for adsorption [31]. To fit more pores into the same volume, there must be a large proportion of micropores [32]. The linear regression for this variable is not quite as good as the surface area, however it is still a fairly strong correlation. Further investigation of this would likely result in stronger correlation than found in this paper. The densities used in this paper were all reported from the manufacturers of the carbon. Measuring density directly for each sample could improve these results even more.

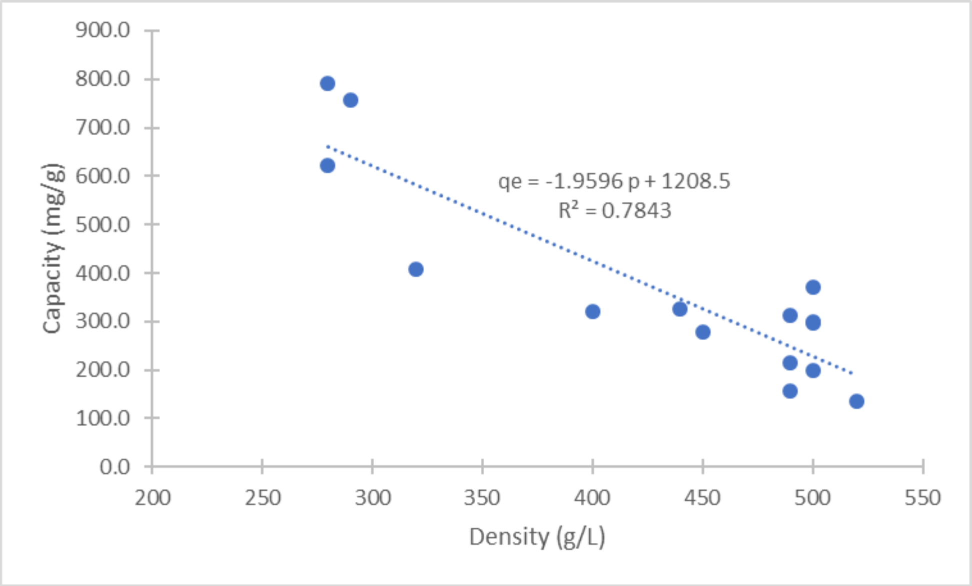


Figure 4: Adsorption Capacity vs Density as Reported

3.2: Adsorption/Breakthrough

Breakthrough is measured in real time for this paper by communications between the PID and the computer. The PID signal has some noise in how it reports, but since each test run takes several hours, the small variations average out. The signal of the PID is also a bit lower than the actual concentration of butanol in the air phase. To correct for this, the weight of several samples was taken, and the concentrations were multiplied by a correction factor to make PID output match the weight of the butanol adsorbed. The correction factor used was between 5.9 and 6.1 for every test. The factor was kept the same for each calibration of the PID, explaining the slight differences.

Breakthrough is defined at 350 ppm as reported by the PID in this paper. Why certain carbons have different shaped breakthroughs will be discussed later, however, the shape of breakthrough does determine when a carbon will reach 350 ppm. An S isotherm with sharp breakthrough will be almost at its maximum capacity before breakthrough. An H isotherm spikes up right away, so a lower breakthrough definition would result in extremely low capacities. Allowing breakthrough to be defined as a value this close to the equivalent concentration ensures that the capacity reported is close to the final capacity for any shape of isotherm.

3.3: Capacity

Each type of AC has a unique breakthrough and capacity due to size, shape, material, and surface functional groups. The summary graph, Figure 2 shows clearly that the wood based carbons have the highest capacity of all other carbons used for this paper. All were provided by Ingevity, and two were extruded and two were granular carbons. The extruded carbons were much smaller in size than the extruded carbons provided by other manufacturers. This helps make a sharper S isotherm, however it doesn’t affect capacity significantly. Upon further literature review, butanol can be derived from wood based sources. It can also be a byproduct of chemical activation of wood, meaning that wood based carbons have a pore structure prone to butanol adsorption [33].

Acid treatment of activated carbon degrades the micropore structure of activated carbon, lowering its capacity for butanol [34]. Butanol is a smaller organic molecule consisting of only four carbon atoms. Without a large proportion of micropores, the butanol does not have as much area to adsorb onto. Typically, it has been found that acid treated carbons have a higher molasses number, indicating that larger molecules have improved capacity [34]. Figure 5 compares the breakthrough of untreated SORBONORIT B4 with the acid and base treated versions of the same original carbon. Not only does the capacity drop significantly, the shape of breakthrough changes completely.

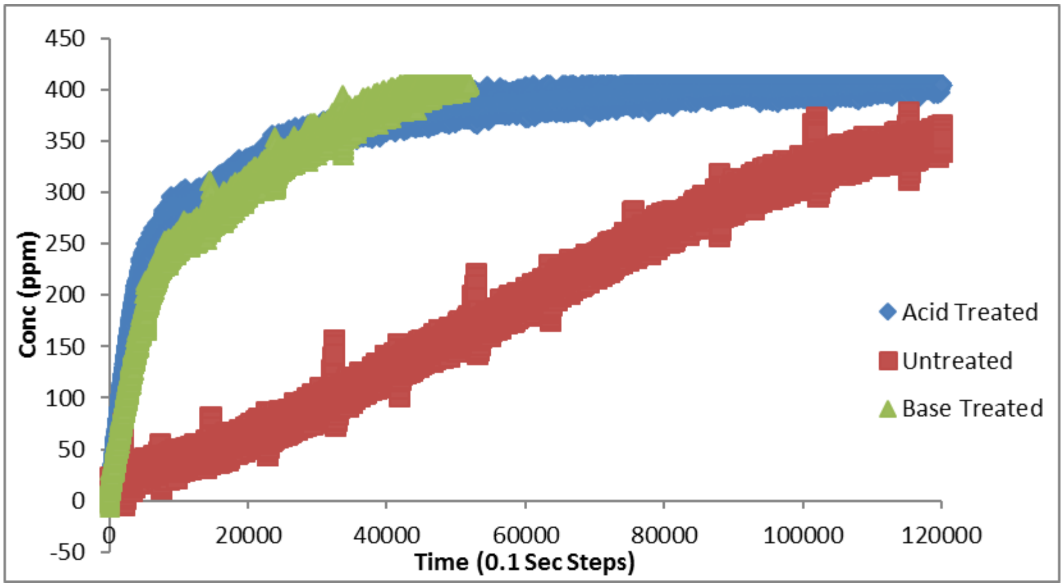


Figure 5: Comparison of Treated vs Untreated SORBONORIT B4

In the case of SORBONORIT B4, capacity drops by about 90%. The destruction of the micropores means there is more empty space inside any given particle of activated carbon. This empty space is not conducive of butanol adsorption, since it is a small molecule, even with multilayer adsorption. The available surface area is therefore much smaller, and the activated carbon is exhausted at a much higher rate.

3.4: Shape of Breakthrough

For most of the carbons tested, butyl alcohol removal via activated carbon adsorption follows the S shaped isotherm. This isotherm has a period of no effluent, and eventually rises to the equivalent concentration before flattening out again, (for single contaminant adsorption) with a clear point of inflection. The other main isotherms are the C, L, and H isotherms, which look like (respectively) a straight line, a function that increases before approaching an asymptote, and an extreme version of the previous shape where the initial slope is extreme and the plateau is sudden [35].

The main benefit of collecting data in real time is the ease of observing isotherm shape. This allows the user to determine whether two tests on the same carbon have been prepared properly. Figure 5 shows two breakthrough curves for NORIT GAC 1240 Plus. Clearly, the shapes of the isotherms are different, even though the adsorbent and adsorbate are identical in both tests. In this case, the S isotherm was due to a tightly packed bed, and the L isotherm was due to a loosely packed bed with channeling through the column.

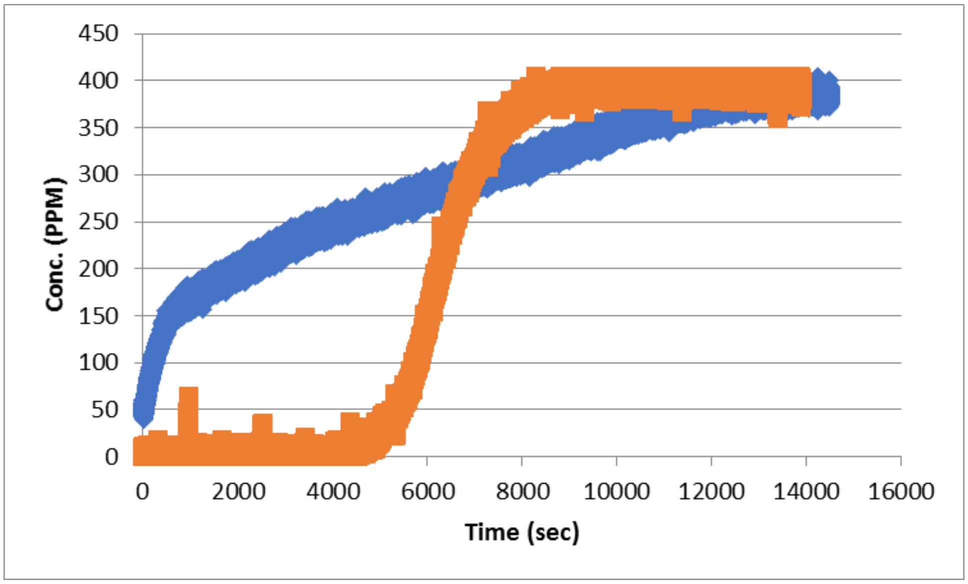
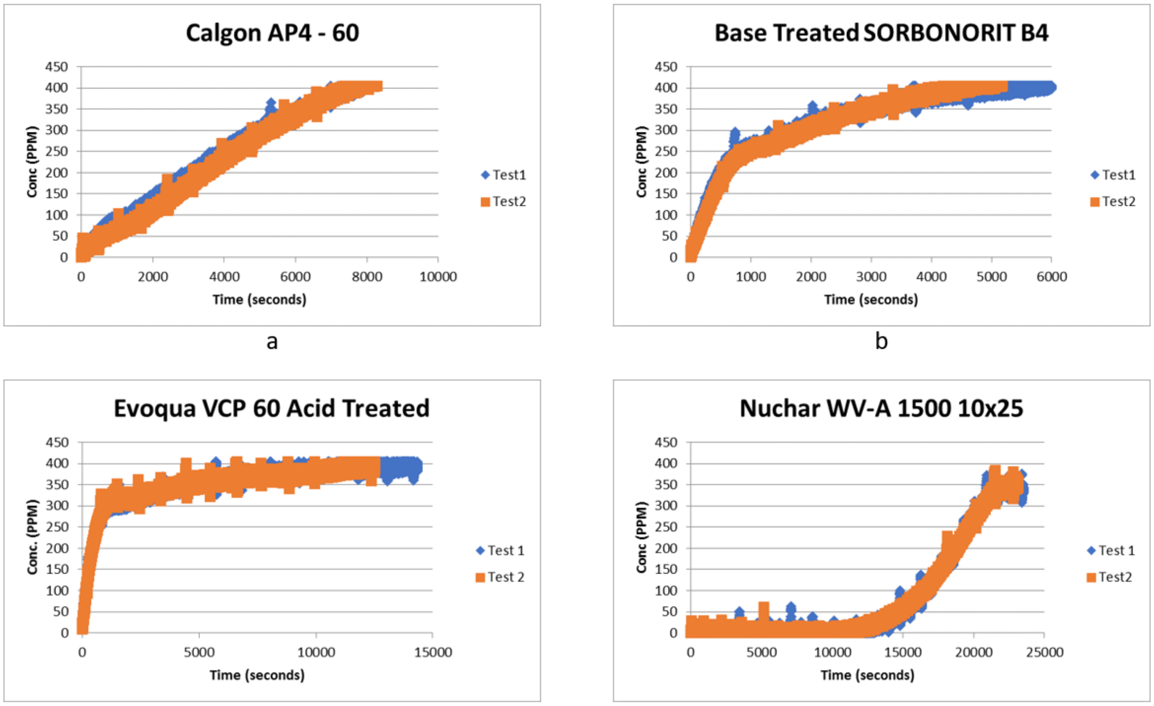


Figure 6: NORIT GAC 1240+ S and L Isotherm Shapes

Each test run takes several hours to complete, and final results are usually quite clear. Within a few minutes however, real time data collection allows drastic changes in isotherm shapes to be noticed. This allows for a test to be aborted early on, before too much time is wasted. Figure 7 shows examples of all four main types of breakthrough curve and which carbon produced that isotherm. Each carbon shows two very similar isotherm shapes, proving repeatability. The respective carbon type is listed above each set of data and each is also labeled with a letter.



a b

c d

Figure 7: Four Main Isotherm Shapes and Repeatability

Figure 7 (a) shows a C isotherm, (b) shows an L isotherm, (c) shows an H isotherm, and (d) shows an s isotherm. Calgon's AP4-60 is the only C isotherm that was observed in this paper. Almost all the treated carbons took on the L isotherm shape as well as some of the larger extruded carbons, with Evoqua VCP 60 being the only H. All other carbons resulted in an S isotherm breakthrough. The isotherm shape is determined by the adsorption kinetics and therefore particle size, surface properties, and pore structure of the activated carbon [25]. Not all tests matched as well as the four shown in Figure 7, which were chosen specifically because of their strong correlation.

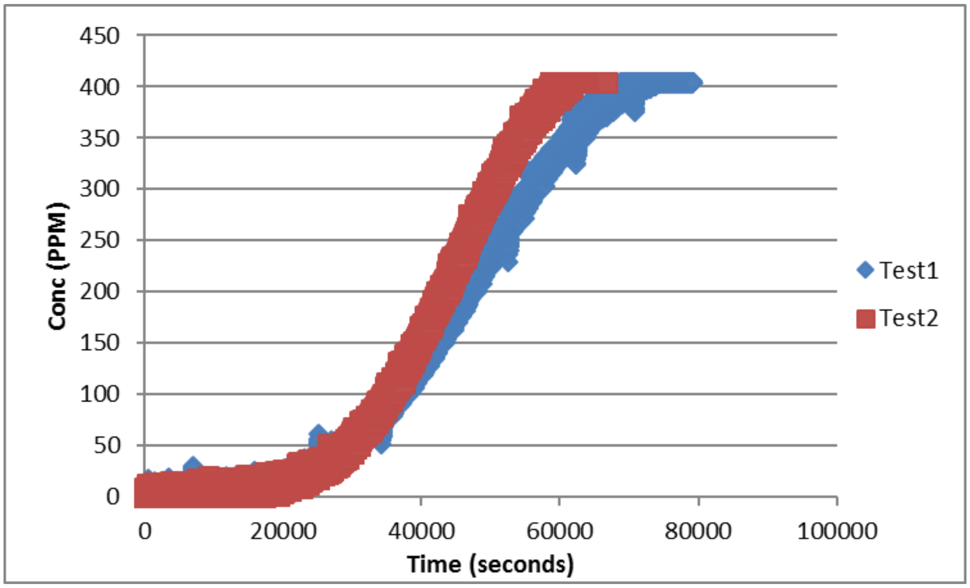


Figure 8: VPR 4X10 Similar Isotherm

Figure 8 shows two tests for Calgon's VPR 4X10 that are similar but do not match as well as the tests in Figure 7. In cases like this, equivalent concentrations were somewhat different for each test. This means that the test with higher concentration didn't take as long, but the total capacities remained almost equivalent. Each test was only included if the capacity was within two standards of deviation. If any test exceeded this, it was excluded and data was collected again. Most were within one normal standard deviation, and excluding tests only happened a few times. Each time one was excluded, something was obviously wrong with the data collection process. Sometimes the packing of the bed was too loose, or other times the flow rate dropped off due to a leak in the tubing. Any leaky tubes were replaced immediately before starting a new test, and results returned to normal.

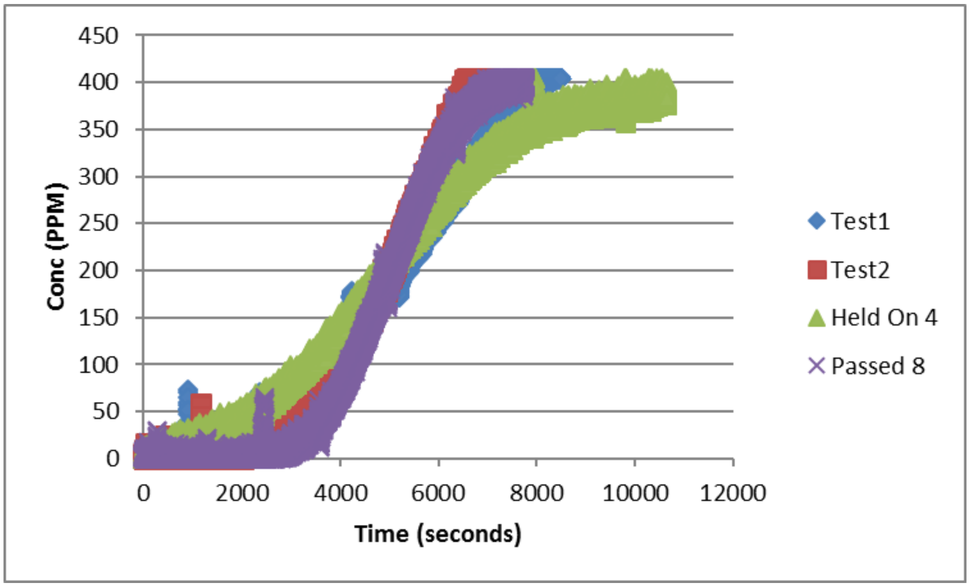


Figure 9: Calgon BPL 4X10 Size Particle Size Distribution

Figure 9 shows four tests for Calgon's BPL 4X10 carbon. Two of them use the bulk material and are almost indistinguishable. The other two consider the particle size of these particles by using sieves to isolate large and small particles. One test was conducted only using large particles that were held on US Sieve #4, and has the least sharp breakthrough. Another test was conducted using only particles that were small enough to pass US Sieve #8. This last test is in the foreground of Figure 9, and has the sharpest breakthrough. All tests gave similar results for capacity, verifying that particle size does not influence results by allowing channeling through the column. BPL 4X10 was chosen for this purpose because it has the largest variety in particle sizes.

4. Conclusion

Using real time data collection, general isotherm shape could be determined after only a few minutes into a multiple hour-long test. This allows the user to determine the quality of the data early on. Using this technique, 15 stock carbons and 6 treated versions were tested for butanol capacity. It was found that acid and base treatment of these carbons reduced butanol capacity by 72 – 91%, due to destruction of the micropore structure. Testing also confirmed good correlations between surface area and capacity, but also supports an inverse relationship between density and capacity. This relationship is likely caused by a well-developed pore structure within the activated carbon particle.

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Appendix: Personal Vita

Dylan Friedgen-Veitch was born to Lori and Scott Friedgen-Veitch on September 23rd, 1993 in Lankenau Hospital near Philadelphia, Pennsylvania. He attended Penn Wood Elementary School, Stetson Middle School, and West Chester Bayard Rustin High School before his college career. He spent one semester at Delaware County Community College before starting at Lehigh University, where he completed his Bachelor of Science degree in environmental engineering in May of 2016. He is tentatively graduating from Lehigh University again in May 2017 with a Master of Science degree, also in environmental engineering. Dylan is a member of the Tau Beta Pi engineering honors society, and is a recipient of Lehigh University's Presidential Scholarship, as well as the Le-Wu Lu Memorial Prize. After graduation, he looks to pursue a career in Environmental Consulting.